

Claims

- [c1] 1. A method of microwave assisted chemistry comprising:
directing a continuous flow of fluid through a microwave cavity while applying microwave radiation to the cavity and to the continuous flow of materials therein;
monitoring the pressure of the fluid in the cavity; and
cooling the fluid in the cavity when the pressure exceeds a predetermined setpoint pressure.
- [c2] 2. A method according to Claim 1 wherein the cooling step comprises moderating the degree of cooling in response to the monitored pressure.
- [c3] 3. A method according to Claim 1 wherein the step of cooling the fluid in the cavity comprises circulating a coolant in the cavity in response to the pressure setpoint determination.
- [c4] 4. A method according to Claim 3 comprising circulating air as the coolant in the cavity.
- [c5] 5. A method according to Claim 1 comprising directing the continuous flow of fluid through a single mode microwave cavity.
- [c6] 6. A method according to Claim 1 wherein the step of directing the fluid comprises directing the fluid in the presence of a catalyst.
- [c7] 7. A method according to Claim 1 wherein the step of directing the fluid comprises directing the fluid in the presence of a scavenging composition.
- [c8] 8. A method according to Claim 1 wherein the steps of monitoring and cooling comprise:
sending a signal representative of the pressure from a pressure monitor to a processor;
using the processor to compare the monitored pressure to the setpoint pressure; and
sending a signal from the processor that initiates and runs a cavity cooling device whenever the monitored pressure exceeds the setpoint pressure.

- [c9] 9. A method according to Claim 1 wherein the steps of directing and cooling the fluid comprise:
directing the fluid through a tube; and
externally cooling the tube.
- [c10] 10. A method according to Claim 9 wherein the step of externally cooling the tube comprises directing a cooling fluid over the exterior of the tube.
- [c11] 11. A method of microwave assisted chemistry comprising
carrying out a chemical reaction in batch format while irradiating the reactants with microwave radiation and while concurrently externally cooling the reaction vessel to thereby identify an optimum power level for the reaction and without exceeding a temperature at which the reactants decompose;
thereafter directing a continuous flow of corresponding reactants through a single mode microwave cavity while applying microwave radiation to the cavity and to the continuous flow of materials therein at the power level identified during batch format reaction of the same reactants; and
externally cooling the flowing reactants while applying the microwave radiation in order to continue at the identified power level while avoiding an undesired increase in the temperature of the reaction.
- [c12] 12. A method of microwave assisted chemistry comprising:
directing a continuous flow of fluid that includes reactants through a single mode microwave cavity while applying microwave radiation to the cavity and to the continuous flow of materials therein; and
purifying the reaction products with a scavenging composition in a single-mode microwave cavity.
- [c13] 13. A method according to Claim 1 wherein the scavenging step comprises directing the fluid through a column filled with a solid support that includes a scavenging functional group selected from the group consisting of electrophilic scavengers, nucleophilic scavengers, and combinations thereof.
- [c14] 14. A method according to Claim 12 and further comprising
monitoring the pressure of the fluid in the cavity; and

cooling the fluid in the cavity when the pressure exceeds a predetermined setpoint.

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| [c15] | 15. A method according to Claim 12 and further comprising:
monitoring the pressure of the fluid in the cavity; and
moderating the applied microwave power when the pressure exceeds a predetermined setpoint. |
| [c16] | 16. A method according to Claim 12 and further comprising:
monitoring the temperature in the cavity; and
cooling the fluid in the cavity when the temperature exceeds a predetermined setpoint. |
| [c17] | 17. A method according to Claim 12 and further comprising:
monitoring the temperature in the cavity; and
moderating the applied microwave power when the temperature exceeds a predetermined setpoint. |
| [c18] | 18. A method according to Claim 12 and further comprising immediately directing the purified reaction products to a separation step. |
| [c19] | 19. A method according to Claim 18 wherein the separation step comprises chromatography. |
| [c20] | 20. A method according to Claim 19 wherein the chromatography step comprises high pressure liquid chromatography. |
| [c21] | 21. An instrument for microwave assisted chemistry comprising:
a microwave cavity;
a flow cell in said cavity;
a pump for directing fluid reactants from at least one source to said flow cell;
a pressure meter in fluid communication with said flow cell for measuring the pressure of fluid in said flow cell; and
a cooling system for cooling said flow cell in said cavity. |
| [c22] | 22. A microwave instrument according to Claim 21 and further comprising:
a processor in communication with said pressure meter and said cooling system |

for moderating the cooling of said flow cell in said cavity in response to the pressure measured by said pressure meter.

- [c23] 23. A microwave instrument according to Claim 22 and further comprising:
a microwave source in communication with said cavity
and wherein said processor is in communication with said source for
moderating the application of microwaves from the source in response to the
pressure detected by said pressure meter.
- [c24] 24. A microwave instrument according to Claim 23 and further comprising a
temperature detector in said cavity and in communication with said processor
for moderating the application of microwaves from said source or moderating
the cooling of said flow cell by said cooling system in response to the
temperature measured by said detector.
- [c25] 25. A microwave instrument according to Claim 21 comprising a single mode
cavity.
- [c26] 26. A microwave instrument according to Claim 21 wherein said flow cell
comprises:
a microwave-transparent support structure; and
microwave transparent tubing on said support structure.
- [c27] 27. A microwave instrument according to Claim 21 comprising a scavenging cell
in said cavity.
- [c28] 28. A method of microwave assisted chemistry comprising:
directing a continuous flow of fluid through a single mode microwave cavity
while applying microwave radiation to the cavity and to the continuous flow of
materials therein;
directing the fluid from the cavity to a spectroscopic flow cell and
spectroscopically evaluating the fluid; and
moderating the conditions in the cavity in response to the spectroscopic
evaluation.
- [c29] 29. A method according to Claim 28 wherein the step of directing the fluid to a

spectroscopic flow cell comprises directing the fluid to a sample line and spectroscopically evaluating the fluid in the sample line.

- [c30] 30. A method according to Claim 28 or Claim 29 wherein the spectroscopy is selected from the group consisting of ultraviolet, infrared and Raman spectroscopy.
- [c31] 31. A method according to Claim 28 wherein the step of moderating the cavity conditions comprises cooling the fluid flow in the cavity.
- [c32] 32. A method according to Claim 28 wherein the step of moderating the cavity conditions comprises adjusting the fluid flow rate through the cavity.
- [c33] 33. A method according to Claim 28 wherein the step of moderating the cavity conditions comprises moderating the microwave power applied in the cavity.
- [c34] 34. An instrument for microwave assisted chemistry comprising:
a microwave cavity;
a flow cell in said cavity;
a spectroscopy cell external to said cavity and in fluid communication with said flow cell; and
a spectrometer with said spectroscopy cell in the optical path of said spectrometer for analyzing the characteristics of fluids flowing from said flow cell and through said spectroscopy cell.
- [c35] 35. An instrument according to Claim 34 comprising a pump in fluid communication with said flow cell for directing fluids from a source and into said flow cell in said cavity;
- 36. An instrument according to Claim 34 and further comprising a system for cooling said flow cell in said cavity during the application of microwaves to said cavity.
- [c36] 37. An instrument according to Claim 34 comprising a single mode cavity.
- [c37] 38. An instrument according to Claim 34 wherein said spectrometer is selected from the group consisting of infrared spectrometers, ultraviolet spectrometers, and Raman spectrometers.

- [c38] 39. An instrument according to Claim 36 and further comprising a processor in signal communication with said spectrometer and with said cooling system.
- [c39] 40. An instrument according to Claim 39 comprising a microwave source in microwave communication with said cavity and in signal communication with said processor; said microwave source being selected from the group consisting of magnetrons, klystrons and solid state devices.
- [c40] 41. An instrument according to Claim 40 comprising a pressure detector in fluid communication with said flow cell and in signal communication with said processor.
- [c41] 42. An instrument according to Claim 40 comprising a temperature detector in said cavity and in signal communication with said processor.
- [c42] 43. An instrument according to Claim 40 comprising a waveguide between said source and said cavity and in microwave communication with said source and said cavity.
- [c43] 44. An instrument for of microwave assisted chemistry comprising:
a microwave cavity;
an attenuator releasably engaged with said cavity and in microwave communication with said cavity;
a flow cell releasably engaged with said attenuator in a manner that fixes the positions of said attenuator and said flow cell with respect to one another when they are engaged and that correspondingly fixes said flow cell in the same position with respect to said cavity when said attenuator is engaged with said cavity.